



## ENHANCING BENDING STRENGTH OF PORCELAIN AFFECTED BY RHA AT DIFFERENT MOULD PRESSURE

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### ABSTRACT

Active silica from rice husk ash RHA was incorporated in a porcelain composition in substitution of quartz. The influence of the substitution on the bending strength, vitrification behavior and microstructure have been investigated. The treated husk was then subjected to calcinations at 700 °C for six (6) hours, after which it was subjected to the XRF analysis. Pellets made with various proportions of kaolin, feldspar, quartz and RHA were pressed at different mould pressures (MP) of 31 MPa - 121 MPa and sintered at a temperature of 1200 °C. Bending strength and porosity testing were carried out to the samples. Samples pressed at 91 MPa with 20 wt% of RHA exhibit maximum bending strength.

**Keywords:** bending strength, mould pressure, RHA.

### INTRODUCTION

Porcelain according to American Society for testing Materials (ASTM) can be defined as a glazed or unglazed vitreous ceramic porcelain made by porcelain process and used for technical purposes, designating such products as electrical, chemical, mechanical, structural and thermal ware when they vitreous [1]. All the various porcelain contain kaolin as the major raw material, i.e., >50 wt%. The other two major raw materials for porcelain products are quartz and feldspar. Quartz, a source of silica, is the most commonly used filler and essential for the microstructural development of porcelain by dissolution of silica in felspathic glass. Quartz is the highest-melting-temperature component in the composition. Quartz grains embedded in the glassy matrix of porcelain have a deleterious effect on the mechanical strength mainly because of  $\alpha$  -  $\beta$  quartz transformation during cooling [2] resulting in the development of stress around quartz grains which initiate fracture [3]. Therefore, researchers attempt to replace it (quartz) by other silica sources that can be used to decrease the processing (sintering) temperature. Among other things used to replace quartz is RHA.

Rice husk ash (RHA) contains a form of silica ( $\text{SiO}_2$ ) and is available in large quantities in Malaysia [4]. Rice husk contains ash from 13 to 29% of weight of the paddy depending on the variety, climate and geographic location [2]. The presence of silica as  $\text{SiO}_2$  in rice husk ash has been known [3] since 1938. The silica in the rice husk is in hydrated amorphous form, either opal or silica gel [5-9]. The use of rice husk ash in the form of silica in the ceramic field was also reported by [6] and [3] also investigated physico-chemical properties of RHA for its application.

Some authors also studied the effect of RHA in porcelain composition [2, 4, 7] and found improvements in the properties with the reduction in the maturing temperature. They have reported that not more than 10 wt% of RHA can be used to substitute quartz. They have also reported a relatively lower densification in porcelain compositions that contain RHA and reactive silica. However, in present study it was found that more than 10

wt% of RHA could be used to substitute quartz in porcelain body.

The porcelain industries make use of natural resources for their production. Increasing world population and life demand are continuously raising the price of raw materials and reducing the natural resources; for these reasons this study is concentrated to use agro-waste materials as potential alternative in the porcelain industry. Keeping this in mind, the present investigation was made to study the effects of substitution of quartz on bending strength of porcelain body (0 - 25 wt%) by RHA in order to help towards sustaining the natural resources, reduce the environmental hazards caused by the ashes and to possibly add value to some of the properties of porcelain.

### EXPERIMENTAL

The RH was thoroughly washed with distilled water in order to remove adhering soil and dust. After that it was dried in an oven at 100 °C for 24 hours. Then the dried husk was subjected to the chemical treatment; 2M HCL, 5% solid at 25 °C before calcinations to increase silica content. After the leaching process, the treated husk was washed with distill water and then dried again. The treated husk was then subjected to calcinations at 700 °C for six (6) hours, after which it was subjected to the XRF analysis. The machine used for the analysis was XRF Bruker S4 Pioneer which was operated at 60 KV and 50 mA.

Porcelain powder was grounded separately in a ball mill. The powder was sieved using 50 $\mu\text{m}$  sieve and dried in an oven. The RHA was gradually incorporated into the body of porcelain powder (Table-1). The composition was mixed using a ball mill for 1.5 hours. The mixed powder was pressed into pellets and bars at pressure of 31 MPa, 61 MPa, 91 MPa and 121 MPa.



**Table-1.** Body composition with replacement of quartz by RHA (wt %).

Mix Number	Kaolin	Feldspar	Quartz	RHA
AP1	50	25	25	0
AP2	50	25	20	5
AP3	50	25	15	10
AP4	50	25	10	15
AP5	50	25	5	20
AP6	50	25	0	25

All the pellets were sintered at the temperature of 1200 °C for 2 hours soaking time, at a heating rate of 5 °C

per minute. The physical and mechanical properties of the pellets such as volume shrinkage, porosity, and compressive strength were determined. The chemical composition of the RHA was studied using x-ray fluorescence (XRF) while the crystalline structure of the pellets was identified through x-ray diffraction (XRD) and the microstructural features were studied by scanning electron microscope (SEM).

## RESULT AND DISCUSSIONS

X-Ray Fluorescence (XRF) analysis is proficient in analyzing material contents inside the raw materials, hence the amount of chemical elements can be observed. The presence of various elements within the raw materials can be seen in Table-2. This table shows the result of XRF analysis of kaolin, feldspar, quartz, and RHA.

**Table-2.** Chemical analysis of the materials.

Sample	Content (% wt)											
	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	FeO <sub>3</sub>	K <sub>2</sub> O	P <sub>2</sub> O <sub>5</sub>	CaO	MgO	CO <sub>2</sub>	SO <sub>3</sub>	Na <sub>2</sub> O	TiO <sub>2</sub>	LOI
RHA	93.70	2.11	-	1.18	0.96	0.81	0.53	010	0.45	-	-	0.16
Kaolin	69.30	24.30	0.27	2.44	-	-	-	0.10	-	-	0.27	0.36
Feldspar	72.70	16.40	0.40	0.50	2.42	-	-	-	6.87	0.29	-	0.10
Quartz	99.40	0.22	-	-	-	-	-	0.10	-	-	-	0.28

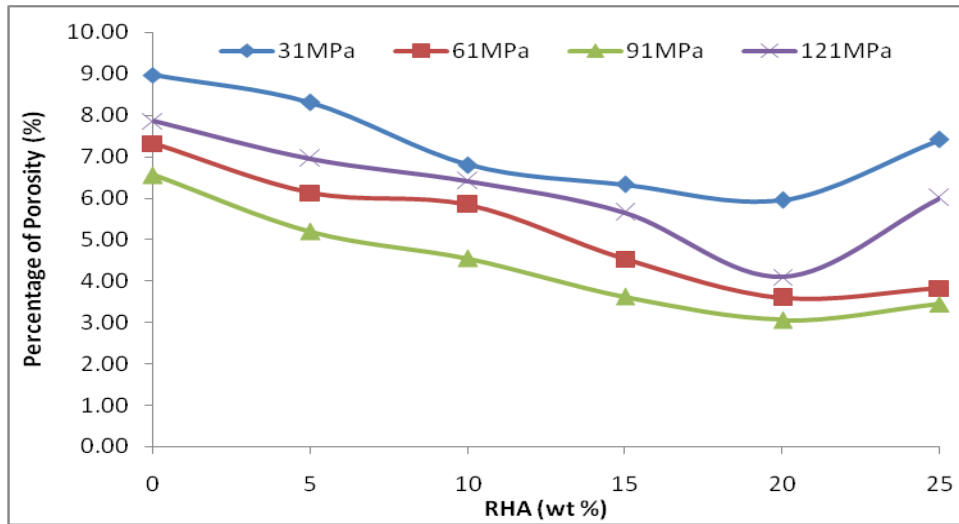
It shows that SiO<sub>2</sub> is the major composition in all the raw materials. There are RHA, kaolin, feldspar and quartz with 93.7 wt%, 69.3 wt%, 72.7 wt%, and 99.4 wt% and followed by alumina with 2.1 wt%, 24.3 wt%, 16.4 wt%, and 0.2 wt%, respectively.

Figure-1 depicts the variation of porosity. The values presented have similar tendency as those previously reported by Perez [10]. The porosity values decreases with increase in substitution of quartz by RHA. The minimum porosity was reached with approximate values of 5.9%, 3.5%, 3.0% and 4.1% on 20 wt% of RHA at the MP of 31 MPa, 61 MPa, 91 MPa and 121 MPa respectively. Similarly, the porosity decreases as the MP increases from 31 MPa to 91 MPa. But as the MP increases to 121 MPa the porosity decreases. Therefore, as the MP increases the capability of water absorption of the samples decreases. However, the porosity increases after reaching minimum as a result of over compaction due to high MP. High MP results in microcracks of the samples.

The porosity values are greater than the value in the literature (0.5%) [10], which is likely due to the differences between industrial and laboratory facilities. The compaction process used in manufacturing porcelain tile typically consist of a double-pressing technology, which involves a first pressing step at very low pressure

values to produce a semi-compact body with a minimum mechanical strength, in which layers of powder decoration are applied. After the pre-compacted body has gone through the decoration processes, it is placed inside a second die for definitive pressing at the traditional porcelain stoneware shaping pressure.

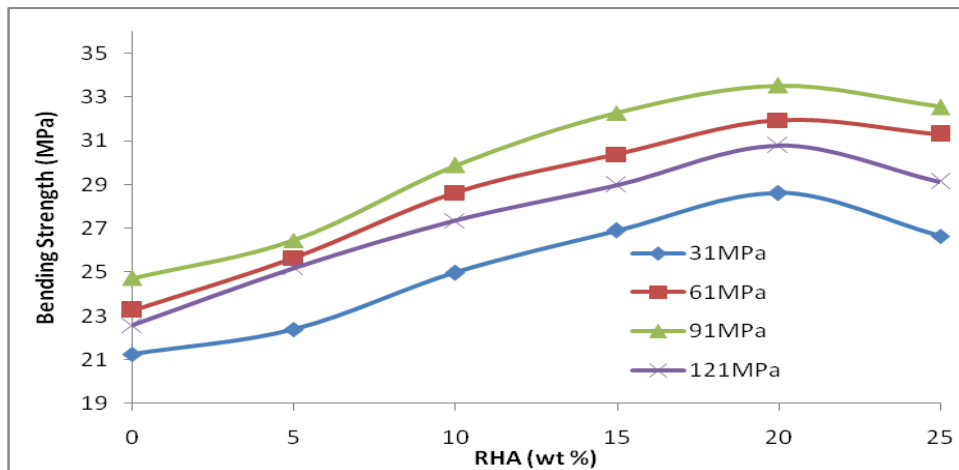
Figure-2 shows the effect of mould pressure on fired bending strength. With the progressive replacement of quartz by RHA. The maximum bending strength was attained with an approximate values of 28 MPa, 31 MPa, 34 MPa and 30 MPa at the mould pressure of 31 MPa, 61 MPa, 91 MPa and 121 MPa respectively with 20 wt% of RHA. The main factor affecting bending strength of samples is porosity. This is because as lower numbers of pores exist, there is less space to contribute to the fracture, since it is known that the relative fracture energy is influenced by pore volume fraction which is the only varying parameter in samples moulded at different pressure. Nevertheless, [11-17] explains that, although porosity has an effect on bending strength of fired samples, there are other factors affecting it, such as mullite formation and quartz particles. This agrees with the findings reported by [18-19]. The bending strength of sample containing 20 wt% of RHA has 36% increase over the standard porcelain ceramic.



**Figure-1.** Effect of MP on percentage of porosity of body mixes with different percentage of RHA.

Consequently, the bending strength increases from 21 MPa to 34 MPa as the mould pressure increases from 31 MPa to 91 MPa. But as the mould pressure

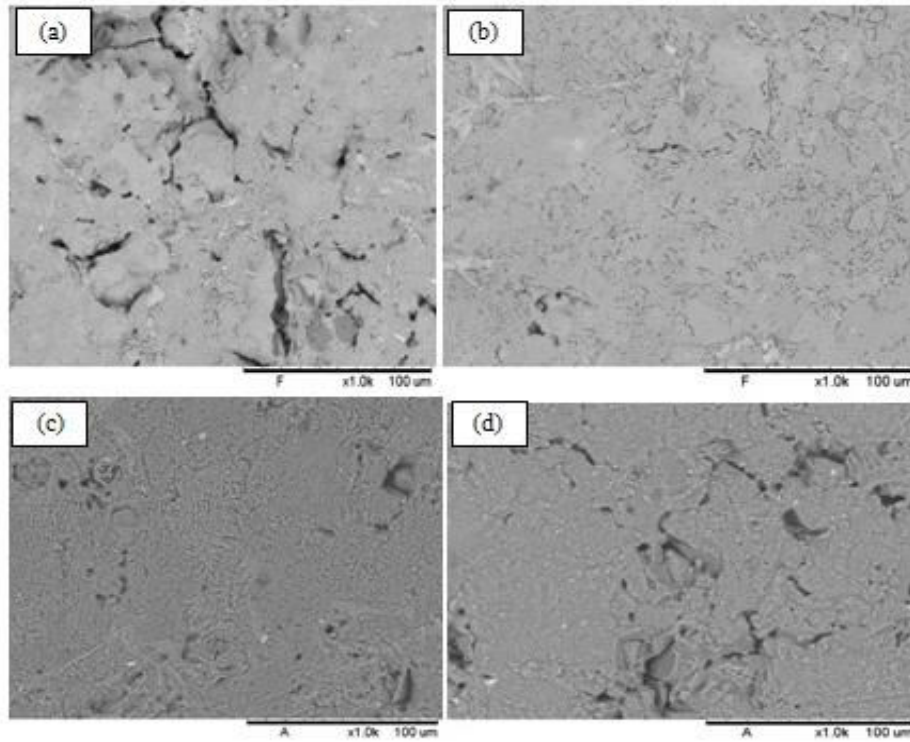
increases to 121 MPa the values of bending strength decreases.



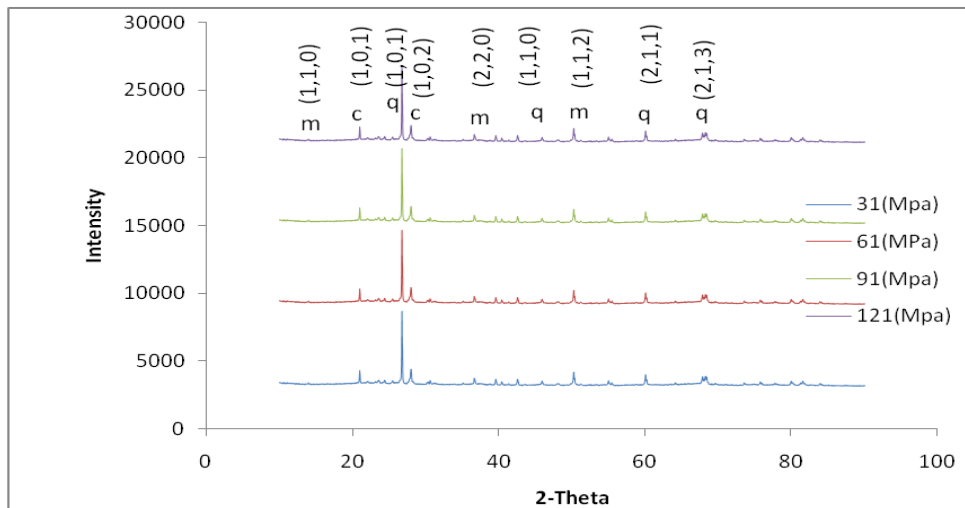
**Figure-2.** Effect of mould pressure on bending strength of the samples with different percentage of RHA.

Figure-3 shows the microstructure of the sintered porcelain samples pressed at different MPs. In the SEM micrographs it can be seen the presence of porosity after sintering. The samples were sintered at a temperature of 1200 °C for 2 hours soaking. At the MP of 31 MPa the porosity consists of interconnected pores with irregular shape (Figure-3a). The porosity decreases as the MP increases to 61 MP (Figure-3b). High MP (91 MPa) leads to a compacted green bodies with lower volume of voids (Figure-4c). At the MP of 121 MPa (Figure 3d) micro-cracks were developed. Similar trend was reported by [20].

XRD patterns of the specimens containing 20 wt% of RHA is shown in Figure-4. The presence of three major crystalline phases namely, quartz (ICDD 046-1045), mullite (ICDD 074-4143), and cristobalite (ICDD 082-0512). In line with the study done by Perez [12] there was not much difference as regard the increase in the phases with MP. The peaks remain almost constant with increase in pressure. Table-3 shows the XRD quantitative analysis of the body mixes, as it can be seen from the table also there is not much difference in the percentages as the MP increases.



**Figure-3.** SEM of the samples sintered containing 20 wt% RHA pressed at MP of (a) 31 MPa (b) 61 MPa (c) 91 MPa (d) 121MPa: All micrographs were taken with 1000X magnification.



**Figure-4.** The XRD curves of sintered the samples containing 20 wt% RHA pressed at different MP.

**Table-3.** XRD quantitative analysis of the samples containing 20 wt% RHA pressed at different MP.

Pressure (MPa)	Quartz (%)	Mullite (%)	Cristobalite (%)	Glassy Phase (%)
31	45.8	35.6	10.5	8.1
61	45.7	36.7	10.5	7.1
91	45.8	36.6	10.3	7.3
121	45.7	36.7	10.6	7.0



## CONCLUSIONS

The bending strength increase with decrease in porosity of the porcelain body as the MP increases from 31 MPa to 121 MPa also with substitution of quartz by RHA. The bending strength reaches maximum when the number of porosity was lowest for samples containing 20 wt% of RHA pressed at the MP of 91 MPa. Microstructure analysis studies reveals that the densification increases with increase in MP. At the soaking time of 2 hours densification takes place least pores were noticed. The increase in the bulk density and the substantial decrease in porosity of the mixes containing RHA, are attributed to the glassy formation and densification of the individual grains during the vitrification process. Both qualitative and quantitative XRD analysis of the body mixes shows not much difference in the percentages as the MP increases.

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